LOW TEMPERATURE EFFECTS ON TNT

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Low temperature effects	Detonation velocity test (miniature)	
DTA	Plate dent test	
DSC	Firing chamber design	
TMA	Coefficient of liner expansion	

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

The effects of low temperature on TNT were determined by subjecting the explosive material to liquid nitrogen (LN_2) and studying the effects through differential thermal analysis, differential scanning calorimetry, thermomechanical analysis, impact sensitivity, detonation velocity and plate dent tests.

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20. Continued

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A coefficient of linear expansion was obtained for both cast and pressed TNT pellets by the thermomechanical analysis method. No permanent changes in thermal stability were observed.

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ABSTRACT

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FOREWORD

This report presents the efforts and results of the Explosives Division, Feltman Research Laboratory, Picatinny Arsenal, in the program studying the effects of low temperature on different properties of TNT. This program was funded by the U.S. Army Mobility Equipment Research and Development Center, Fort Belvoir, Virginia under AMC Form 1095 P/WD dated 12 July 1973. The technical monitor for the program was Dr. David C. Heberlein.

Introduction

1. Purpose of the Program

The purpose of this program was to determine the effects of low temperature on TNT. The possible use of liquid nitrogen in mine applications has generated a need for data regarding the explosive performance of TNT in that type of environment. This information will enable extrapolation of expected behavior for particular mine types as well as buried or surface implanted mines.

2. Scope of Work

The program consisted of testing, analyses and evaluation of the effects of a cryogenic environment on the explosive properties of TNT. The specific tasks originally outlined when the program was proposed included the following:

a. Preliminary calorimetric studies to identify possible solid phase transformations occurring at low temperatures.

b. Experimental measurements to determine the sensitivity of TNT to impact at low temperatures.

c. The detonation rate f(t) of TNT both confined and unconfined in cast and pressed form.

d. Changes in explosive yields or outputs as a function of temperature. Minimal booster sizes to effect detonation also were to be determined.

e. The data for both confined and unconfined, pressed or cast, TNT to be examined for promising areas of application.

Low Temperature Experimental Procedures and Results

All the data reported here were obtained with TNT (2,4,6-trinitrotoluene) manufactured according to military specification MIL-T-248A, Amendment 1, Type 1, flake (Lots KNK-11-364 and VA 20-214, set point 80.20°C). All the experiments were performed in a cryogenic environment using liquid nitrogen (LN_2) and under ambient conditions for comparison purposes. Commercial TNT contains from 98% to 99.5% of the 2,4,6-isomer with the purity usually being specified in terms of a setting-point.

1. Thermal Stability

The effect of LN_2 on the thermal stability of TNT was determined from results of the differential thermal analyses tests, differential scanning calorimetry tests and the thermomechanical analysis tests. Efforts were made to determine if any possible solid phase transformations were evident.

a. Differential Thermal Analysis (DTA)

Differential thermal analysis is a technique by which the thermal effects associated with physical and chemical changes are recorded as a function of temperature

as the substance studied is heated at a controlled rate. With this technique the temperature of the sample is compared continuously with that of an inert reference sample and the difference in temperature is recorded as a function of the heater temperature. The record obtained is the DTA curve or thermogram, and, if the material is thermally active, the thermogram indicates a series of peaks. The position of these peaks is determined by the chemical composition and crystal structure of the material and the area is related to the reaction occurring in that temperature range.

For this program the DTA curves were obtained using the duPont Differential Thermal Analyzer, Model 900, with a remote cell adapter which was modified so that it could be immersed in LN_2.

b. Differential Scanning Calorimetry (DSC)

Differential scanning calorimetry (DSC) is closely related to differential thermal analysis (DTA). It measures the electrical energy input necessary to establish zero temperature difference between a substance and a reference material against either time or temperature as they are both subjected to a controlled changing temperature. The DSC varies the amount of heat added to the substance and the reference material in order to maintain them at the

same temperature and measures the difference in an electrical power in calories per second.

The thermal properties that can be found with the reduction of the DSC thermograms include: melting point, purity of the compound, activation energy, specific heat and solid state transition energies.

In this study the Perkin-Elmer DSC-1B Differential Scanning Calorimeter, a Mosley Autograph Model 7100A Recorder and a Cahn Gram Microbalance were used.

c. Thermomechanical Analysis (TMA)

The TMS-1 Thermomechanical Analyzer provides a record of dimensional and viscoelastic changes in a solid-state sample heated in a standard furnace from -150°C to 325°C (123 to 598K). Temperature programming is provided by the Perkin-Elmer UU-1 Temperature Program Control (Fig. 1). The Model TMS-1 is a device which can measure with extreme sensitivity the linear displacement of a movable probe relative to a fixed point.

The principal quantitative calculation determined from the TMA thermogram is the coefficient of linear expansion. Also a glass transition is evident by a discontinuous change in the coefficient of expansion. First order transitions (crystal-crystal, melting, etc.) show a characteristic behavior of TMA. The coefficient of expansion was required in order to correct the density of the TNT

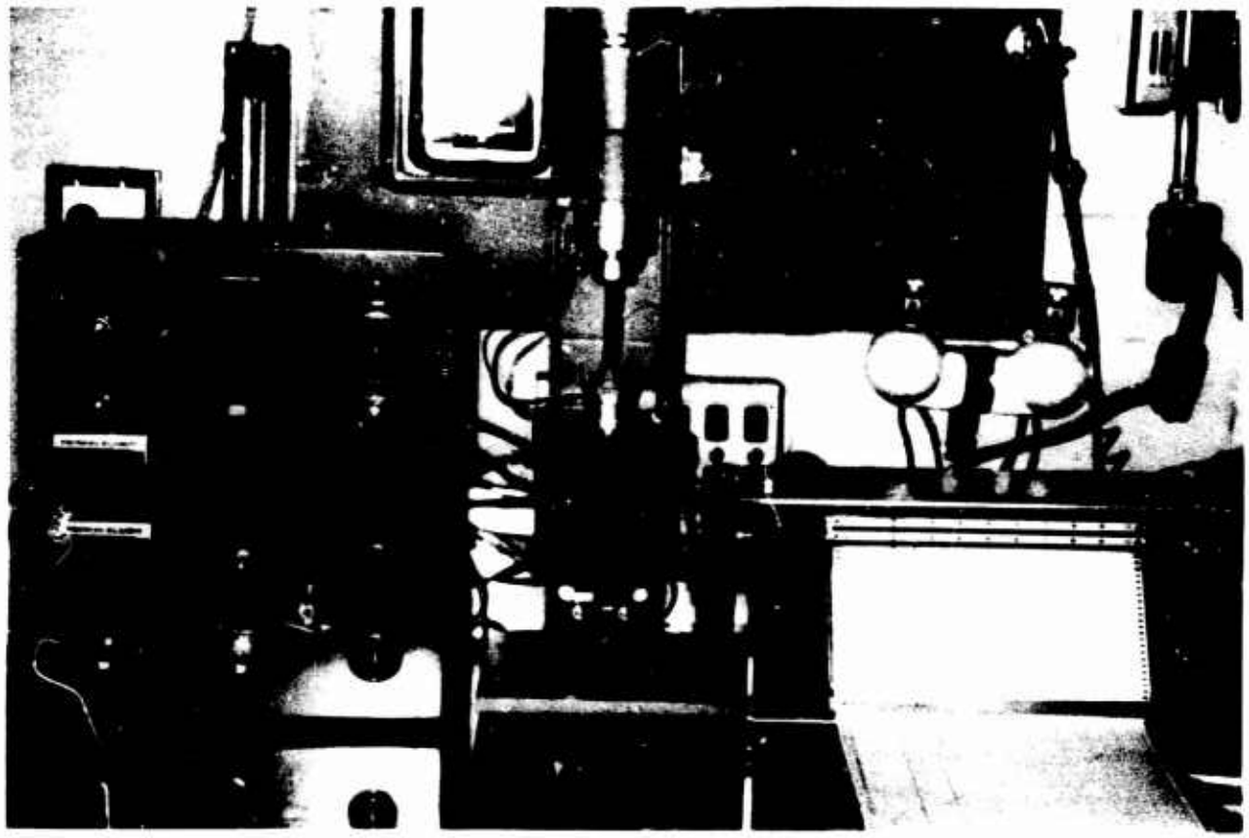


FIGURE 1. Thermomechanical Analyzer Equipment (TMA).

pellets and the detonation velocity values from ambient to LN_2 conditions.

2. Sensitivity

a. Impact Sensitivity

The standard Picatinny Arsenal impact sensitivity test (1) was modified (2) in order to determine the effect of low temperature on the impact sensitivity of TNT. The modifications had been used in another program (3) where the effects of LN_2 were determined on a variety of explosives and explosive mixtures. In order to conduct the impact test in LN_2, solid steel cylinders 2" in diameter and 1 1/2" in height were machined as receptacles for the LN_2. From each of the cylinders a well 1 3/4" in diameter and 1" deep with an inset to hold the die cup, 0.350" in diameter and 0.125" deep, was reamed out (Fig. 2). A steel guide slipped over the top of the well. The guide had a hole in the center which aligned and permitted a 3/8" punch 3 3/4" in length to rest on the impact test fixture encasing the explosive.

The impact test fixture consisted of a die cup, a brass cap and a vented plug. The explosive was loaded filling the die cup and the brass cap covering the die cup. The assembly was placed in the inset of the well. The vented plug sat atop and centered on the brass cap.

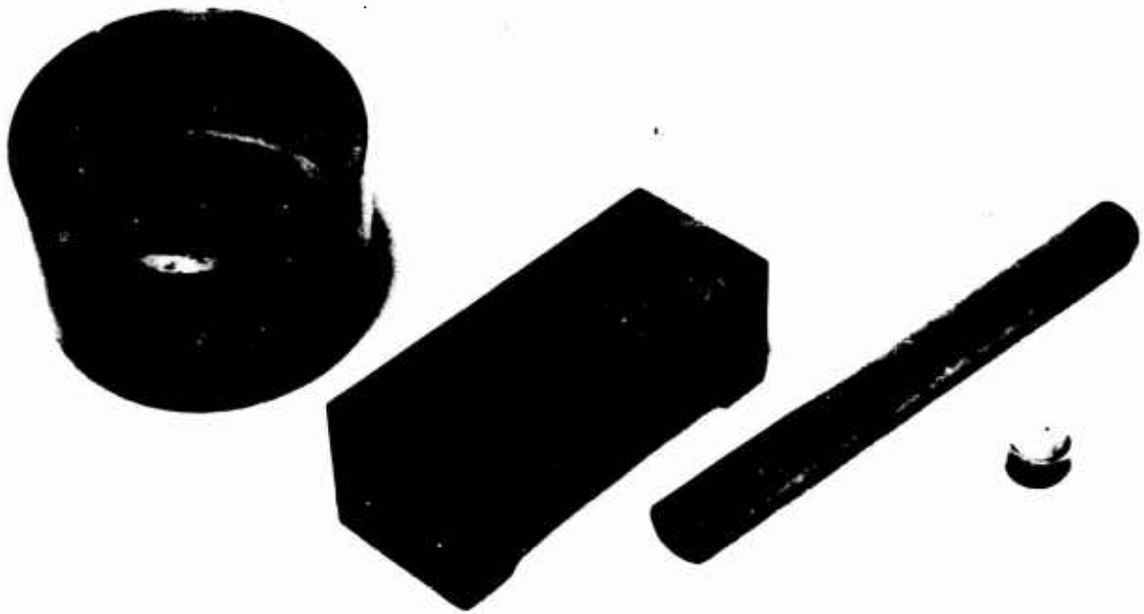


FIGURE 2. Well, Guide and Punch for Impact Test.

The 3/8" punch rested on the vented plug and the drop weight of the impact tester was dropped onto the punch from predetermined heights (Figs. 3,4).

Before conducting the impact test in a cryogenic environment, measurements were made to determine the time of immersion in LN_2 for the impact test fixture to reach thermal equilibrium. The average time for cooling from ambient to LN_2 temperature was 3.1 minutes. A cooling time of 4 minutes was selected for the subsequent tests to insure thermal equilibrium.

Normally the loaded die cups were kept in a tray immersed in LN_2 for about 10 minutes before being transferred to the steel well. Upon placing the loaded fixture in the inset, the steel well was filled with LN_2 from a Dewar. Each sample, which was immersed in LN_2 while in the well was tested after an equilibrium period of 4 minutes.

3. Explosive Performance

a. Miniature Detonation Velocity Measurement

Detonation velocity is one of the most important measurements of explosive performance. Large scale detonation velocity measurements have been made for many years, and still have the highest accuracy. However, with limited quantities and space, hazardous explosives and/or

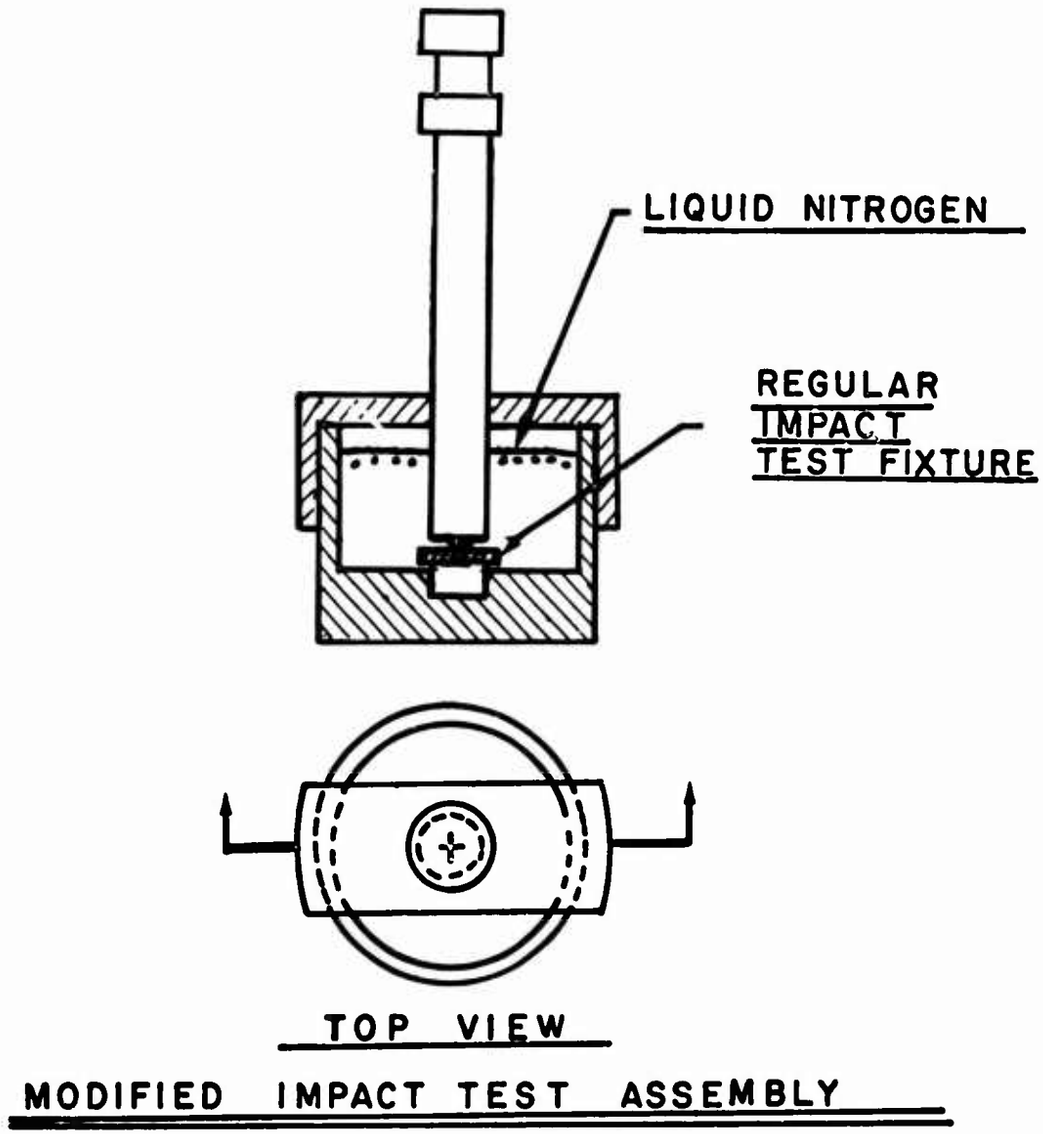


FIGURE 3. Sketch of Modified Impact Test Assembly.

FIGURE 4. Picatinny Arsenal Impact Tester with Cryogenic Fixture.

limited funds it is desirable to obtain a measurement on smaller samples. The following procedures were developed for use with samples of 1/2" diameter and smaller with sample weights less than 15 grams.

(1) Distance Measurement

For small samples the measurement of distance between switches and of time intervals must be as precise as possible. Distance of pin locations are measured by a Gaertner M303A travelling microscope accurate to 0.0001 inches and pellet lengths with a micrometer having the same accuracy. Time intervals are measured to an accuracy of $\pm$ 5 nanoseconds (ns) or better.

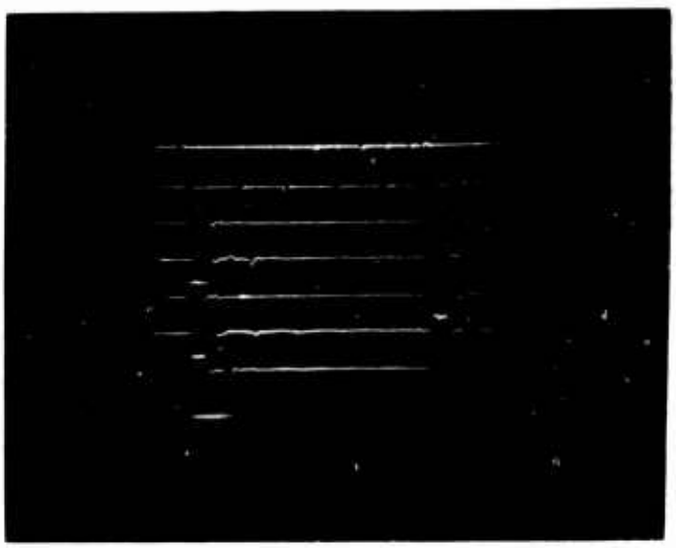
(2) Time Measurement

Three methods can be used - (1) time interval meter (Eldorado 784 or 793), (2) Raster oscilloscope (Tektronix 535A with Cordin 1136 plug in or Moran 101 raster system), and (3) transient digital recorder (Biomation 8100). Generally, it is desirable to use at least two systems simultaneously. The type of contact made by the detonation reaction is not always the same, and therefore rise times vary. The time interval meter has the disadvantage that the trigger levels must be set befo· the test, and so errors can be introduced due to these variations.

The preferred method of data recording is the use of the Biomation 8100 recorder. This recorder has a rise time of 15 ns, and samples the analog data every 10 ns. The voltages are then converted into a digital number (one part in 256) which is stored in a 2000 number memory. This gives a total recording time of 20 microseconds, which is more than adequate for the size of samples used. After the data is recorded, it can be re-read in analog form, as shown in Figure 5, with portions of the record expanded for more accuracy as shown in the lower portion of the record. However, the preferred method is to transfer the numerical data to magnetic tape, and then read and process it by computer.

(3) Precision of Detonation Velcoity (D) versus Precision of Time Measurement (t)

The question has been raised many times what constitutes preciseness in determining the detonation velocity of an explosive. An analysis was made utilizing the geometry of the detonation velocity test used in this program. However, it is general in scope to be applied to any similar test to either compute the precision of the test as a function of the time measurement, or to decide what time measurement precision must be realized to achieve a chosen test precision with respect to the time parameter.



Pressed TNT Pellets, 3/8" Dia., Brass Confined

Tested 10/1/74

Top Trace	2μs/cm
Second Trace	1μs/cm
Last 5 Traces	0.2μs/cm

FIGURE 5. Detonation Velocity Record in Analog Form.

Since $s = vt$ or $v = s/t$

the differentiating

$$\frac{\partial v}{\partial t} = -\frac{s}{t^2}$$

or as s is a constant (i.e. has no uncorrectable error)

then $$dv = -\frac{s}{t^2}\,dt = -\frac{v}{t}\,dt \qquad (1)$$

Equation (1) may be used to calculate the absolute error in velocity, dv, as a function of the time error, dt. Then the relative error may be drived from

$$\frac{dv}{v} = -\frac{dt}{t} \qquad (2)$$

From this it can be seen that the relative error decreases as the time increment increases with a fixed time error.

For example, assume the irreducible timing error dt is 10 ns and it is not a function of the length of time measured. That is the case, generally, with pin switches. The error is generated only by the precision with which the start and stop signals are read, the intervening time having an error one or more orders of magnitude smaller because of markers, high-precision oscillation, etc. The 10 ns value is almost the same for the specified precision of the Biomation recorder and is also what is usually obtained with careful oscillogram analysis.

For explosive material having a detonation velocity, D = 6 mm/μsec the error in velocity for a segment 3/8" (9.5 mm) long, which takes about 1.59 μsec to transverse. is

$$dv = \frac{6}{1.59}\ (0.010) = .038 \text{ mm/μsec}$$

which is ∿ 1/2% of the D = 6 mm/μsec

At D = 8 mm/μsec

$$dv = \frac{8}{1.19}\ (0.010) = .07 \text{ mm/μsec}$$

or ∿ 1.0% of D.

However lengthening the segment measured, say 1.875" (47.3 mm) from 3/8" (9.5 mm) (the true traversed distance from the first pin after t_o to the last pin), with D = 6 mm/μsec the time to traverse the distance travelled is 7.88 sec then

$$dv = \frac{6}{7.88}\ (0.010) = .008 \text{ mm/μsec}$$

or if D increases to D = 8 mm/μsec

$$dv = \frac{8}{5.91}\ (0.010) = 0.13 \text{ mm/μsec}$$

which would result in errors of 0.13% and 0.16% respectively.

b. Explosive Sample Preparation

The TNT explosive samples were prepared in several ways in order to determine the effect of LN_2 on the detonation velocity of that explosive. Basically the TNT pellets were pressed or cast and tested in a confined or unconfined manner. In another instance the TNT was pressed in increments in an aluminum tube.

The pressed pellets were made in two sizes. The 1/4" (.625 cm) OD pressed TNT pellet was nominally 0.375" (.753 cm) in length, 0.40 grams (gm) in weight and 1.60 gm/cc in density when pressed at 40,000 psi (loading pressure 1 ton). The 1/2" (1.27 cm) OD pressed TNT pellet was 0.72" (1.83 cm) in length, 3.6 gm. in weight and 1.61 gm/cc in density when loaded at 30,000 psi (3 ton loading pressure).

The cast pellets were much simpler to make the 1/4" OD cast TNT pellet was 1" (2.54 cm) in length, 1.30 gm in weight and 1.60 gm/cc in density. The 1/2" OD cast TNT pellet was 0.80" (2.03 gm) in length, 40 gm in weight and 1.58 gm/cc in density.

The TNT pressed in an aluminum tube .356" OD (.904 cm) .319" ID (.81 cm) and 3" long (7.62 cm) was done in three or four increments at 25,000 psi (loading pressure 1 ton) with a density of 1.624 gm/cc.

c. Lightly-Confined Detonation Velocity Test on 1/4" Diameter Explosive Charge

The smallest explosive samples tested in this program were 1/4" diameter. TNT pellets 3/8" long were pressed or cast, and then stacked in aluminum tubes having 0.028" wall thickness. The test explosive column was 1 1/2" long, and was initiated by a detonator and a 1/4" long tetryl booster pellet. A mild steel witness plate, 1 1/4" OD and 1/8" thick was attached to the base of the column as shown in Figure 6. Velocity was allowed to stabilize over the first 1/2" and measured over the last 1" of distance. The space between the explosive pellets and the inner tube wall was 4-7 thousands of an inch and was filled with silicone grease. Electrical signals were obtained from enameled copper wires (#30 gage or 0.014" dia.) glued into 0.016" diameter holes drilled in the side of the tubes.

To measure the time of travel a time interval meter was incorporated in the pin switch start-stop circuit as shown in Figure 7. As the detonation wave passes each pin switch it generates a pulse which is picked up by the timer. The interval between the two pulses is the time of travel for the detonation wave between the two pins. When more than two signals were to be recorded the multiple signal

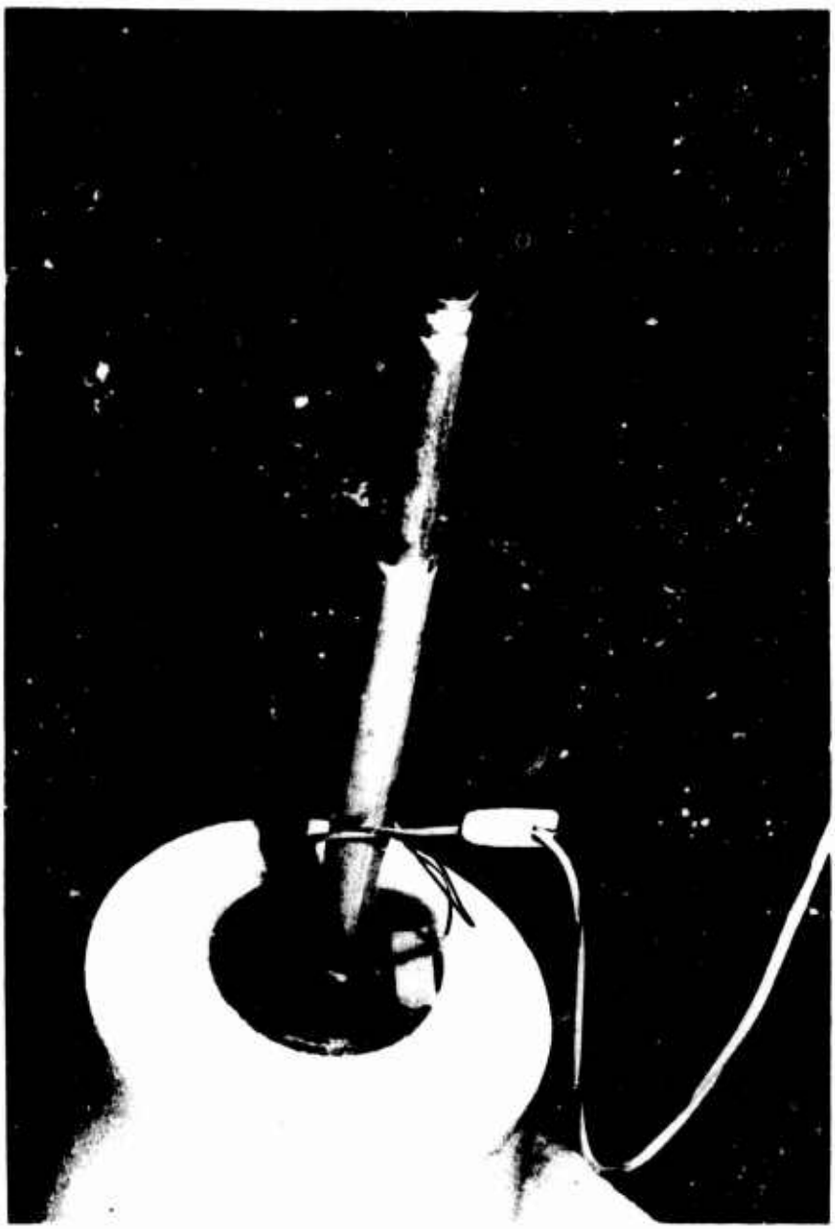


FIGURE 6. Lightly-Confined Detonation Velocity Test (Two-Point Measurement)

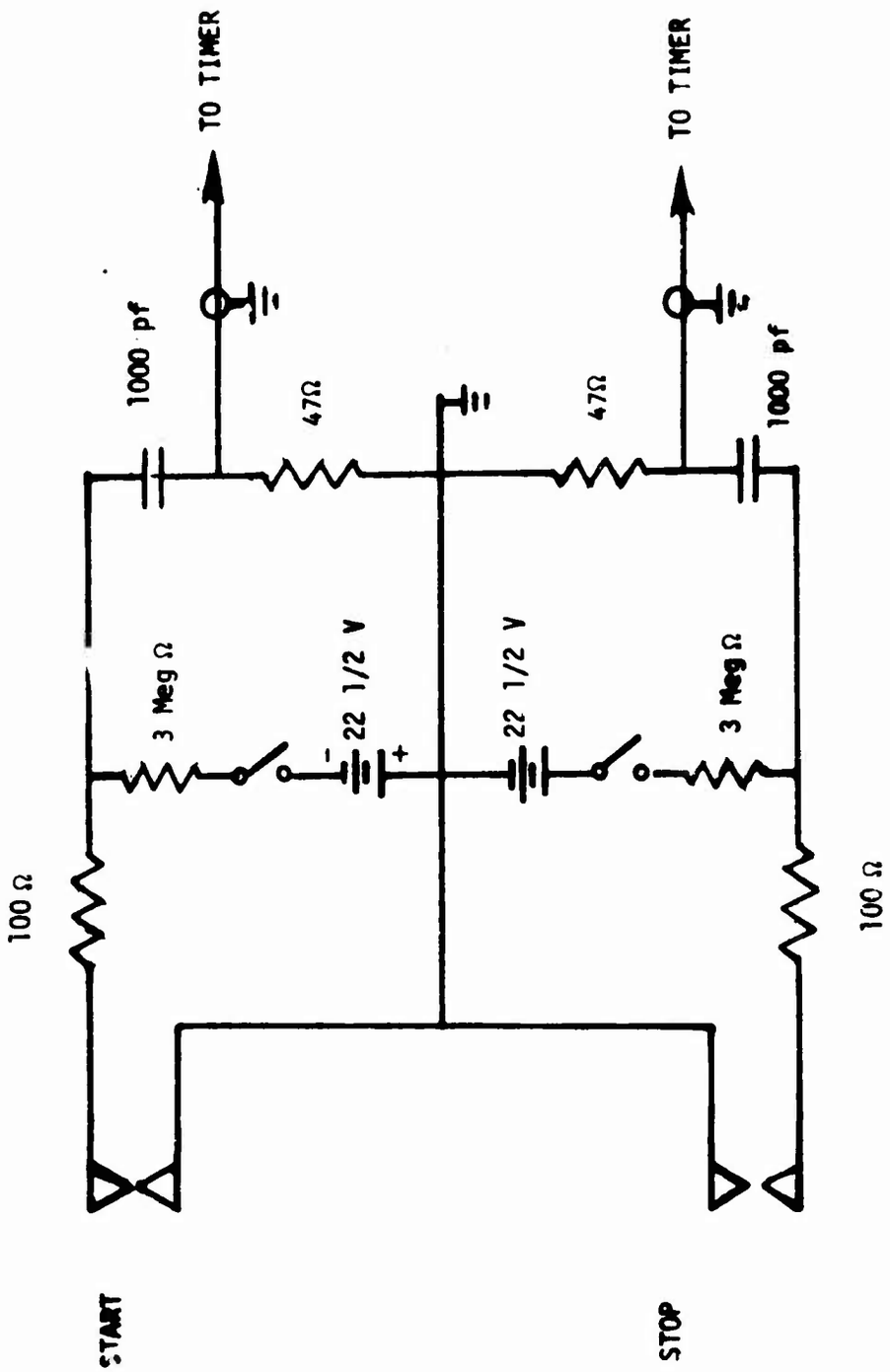


FIGURE 7. Start-Stop Pin Switch Circuit.

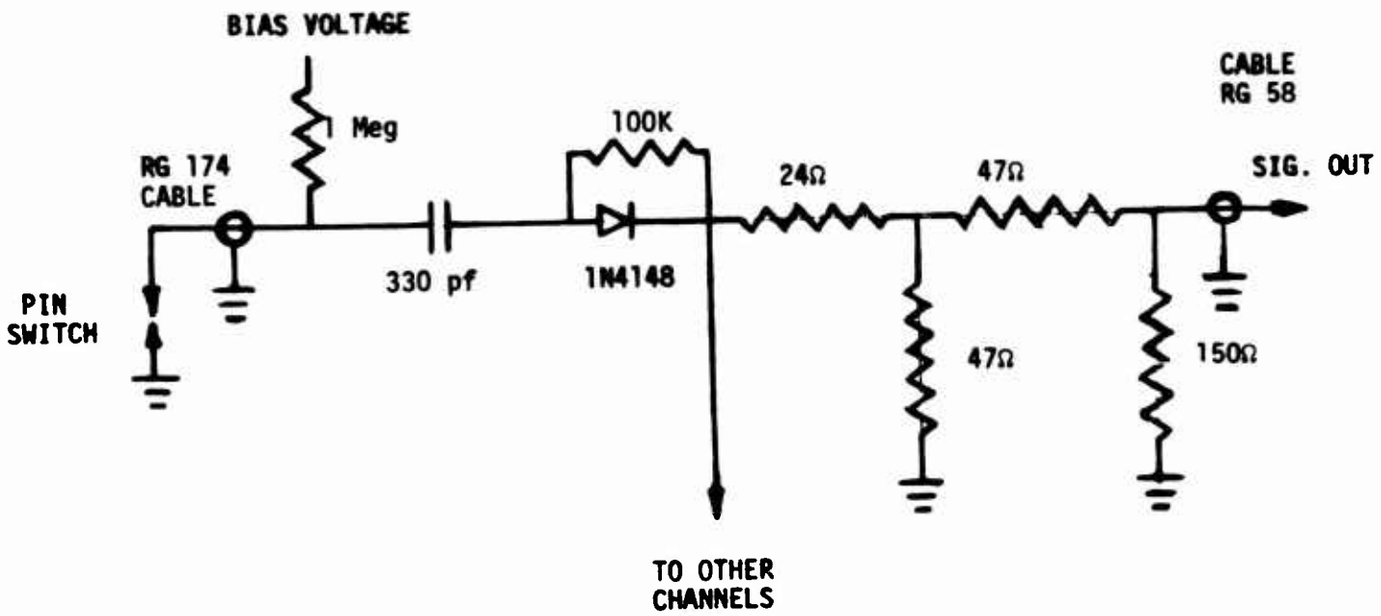


FIGURE 8. LLL Multiple Pin Switch Circuit.

mixer pin switch circuit shown in Figure 8 was used which produced traces as depicted in Figure 5.

In order to determine the overall accuracy and reliability of the technique, a series of 24 tests were conducted using 9404 (an explosive consisting of 94% HMX, 3% nitrocellulose, 3% chloroethyl phosphate) pressed and machined explosive pellets. The 9404 pellets were loaded and tested in the same configuration as Figure 6 except five pin switches were used instead of two for the same distance in order to obtain more data. The results are listed in Table 1.

The average velocity was 8763.3 m/sec with a standard deviation of 115.4 m/sec for the 24 tests. The average velocity agrees with the 8754 m/sec value for the same dimensions and density reported by LLL. Based on the standard deviation, the measured velocities are expected to be accurate within 2.6% at a 95% confidence level. Larger samples, of course, provide a higher precision.

d. Heavily-Confined Detonation Velocity Test

At the time of this program another effort was made to obtain detonation velocities on explosives materials which required a heavily-confined casing. Therefore tests were conducted in a configuration to

TABLE 1

Detonation Velocity Data on Pressed 9404 Charges
(9404 Explosive Cylinders 1/4-in Diameter
Confined in Aluminum Tubes)

Test No.	9404 Density (gm/cc)	Velocity Between Individual Points, m/sec 1-2	2-3	3-4	4-5	Points Included in Average	Average Velocity (m/sec)
1	1.844	8504	8710	9796	-	1-4	8873.9
2	1.840	9051		8643	8566	1-5	8838.3
3	1.843		8515	7841	9559	2-5	8681.9
4	1.843		8654	9003		2-5	8868.7
5	1.839	8877	9207	8014	8224	1-5	8606.9
6	1.842	8763	8036	95.7	8799	1-5	8633.0
7	1.842	8776	8782	8159	9660	1-5	8877.1
8	1.842	8986	8697	9005	8964	1-5	8903.8
9	1.843	8514	8716	8410	8759	1-5	8643.5
10	1.840	8992	8322	9360	9156	1-5	8903.4
11	1.839	-	8646	9276	8190	2-5	8611.5
12	1.840	9100	8414	8657	8433	1-5	8775.9
13	1.839	8210	9134	8012	9197	1-5	8663.7
14	1.843	9350	8219	9778	8852	1-5	8947.4
15	1.842	8720	9095	8512	8748	1-5	8786.9
16	1.846	8472	8701	9005	8750	1-5	8703.7
17	1.844	8734	9114	8340	8194	1-5	8603.7
18	1.840	8964	9289		8372	1-5	8933.8
19	1.842	9052	8957	8454	8594	1-5	8786.3
20	1.841	8610	9055	8498	2918	1-5	8867.0
21	1.841	8858	8475	8245	8781	1-5	8615.4
22	1.839	8611	8821	8536	8639	1-5	8661.1
23	1.840	9124	8669	8312	8595	1-5	8703.3
24	1.841	8893	8983	8671	8552	1-5	8780.8
Average	1.8415						8763.3
Standard Deviation, m/sec							115.4

determine the effect of thick walls on the velocity in a LN_2 environment. A higher energy booster pellet (9404-94% HMX/3% NC/3% CFP) was also used to improve initiation.

Heavily confined TNT samples, 0.319" in diameter, were fired in 1" OD brass sleeves as shown in Figure 9. The aluminum sleeve, 3/8" OD and 0.319" ID with 0.028" wall thickness, was anodized to electrically insulate the switch wires until the anrival of the shock wave. Since the samples were electrically insulated from the tube, hazards due to electrical potential on the pin switches were eliminated. The tubes also facilitated accurate density measurements, and protected the samples from moisture or physical damage.

As indicated previously the TNT was pressed in the 3/8" tube in several increments at 25,000 psi giving a density of 1.584 gm/cc. The 3/8" aluminum tube was placed into the brass tube topped by a 9404 booster pellet and a RP87 EBW detonator. The whole assembly was bonded to a steel witness disc, 3/4" thick and 1 1/2" diameter.

For LN_2 tests this entire assembly was placed in a paper cup filled with LN_2 up to the detonator and allowed to cool for at least 5 minutes.

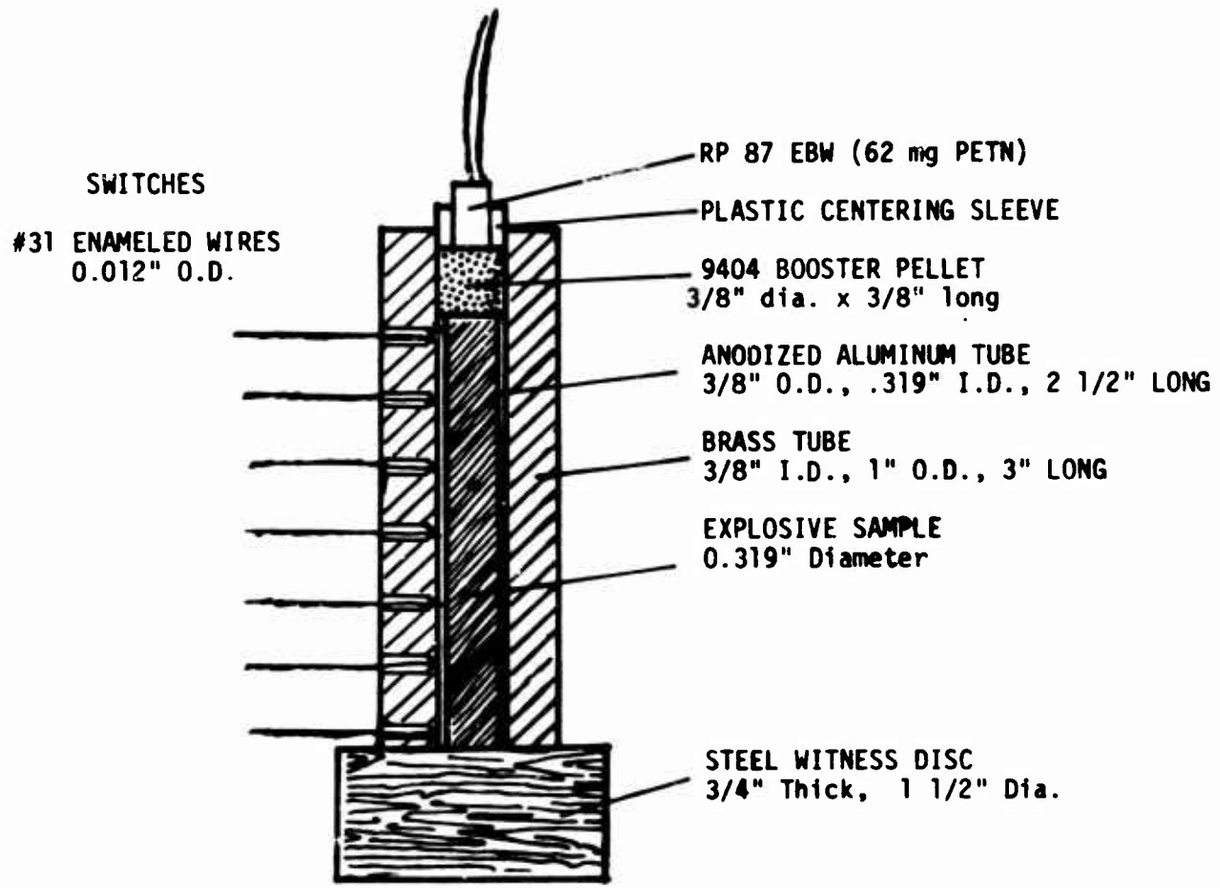


FIGURE 9. 3/8" Diameter Heavily Confined Detonation Velocity Test.

e. Unconfined Detonation Velocity Test

Unconfined TNT explosive pellets, 1/2" diameter, and 3/4" long, were stacked up in a column as shown in Figure 10, and clamped into position on top of a steel witness block. The springs on the rods between the lucite block and nuts were to keep tension on the pellets which contrasted when immersed in LN_2. Measurements are desired of the dent depths in order to determine the total impulse, particularly if non-ideal or heterogeneous explosives are being tested. The hardness and composition of the steel blocks must therefore be controlled. Ionization-type foil switches, reaching half way across the diameter of the charges were used to detect passage of the detonation wave. The lead wires being directed away from the source of the detonation, or shielded to prevent premature failure of the sensing circuit. A sketch of the set-up when immersed in LN_2 is shown in Figure 11.

Firing Chamber Designed and Set-Up

In order to conduct the "miniature detonation velocity test" in a limited space, i.e. in the hood of a laboratory, a firing chamber was designed to fully contain the explosion and the metal fragments from the explosive holder and detonator. Using the experience gained in designing explosive containment capsules for

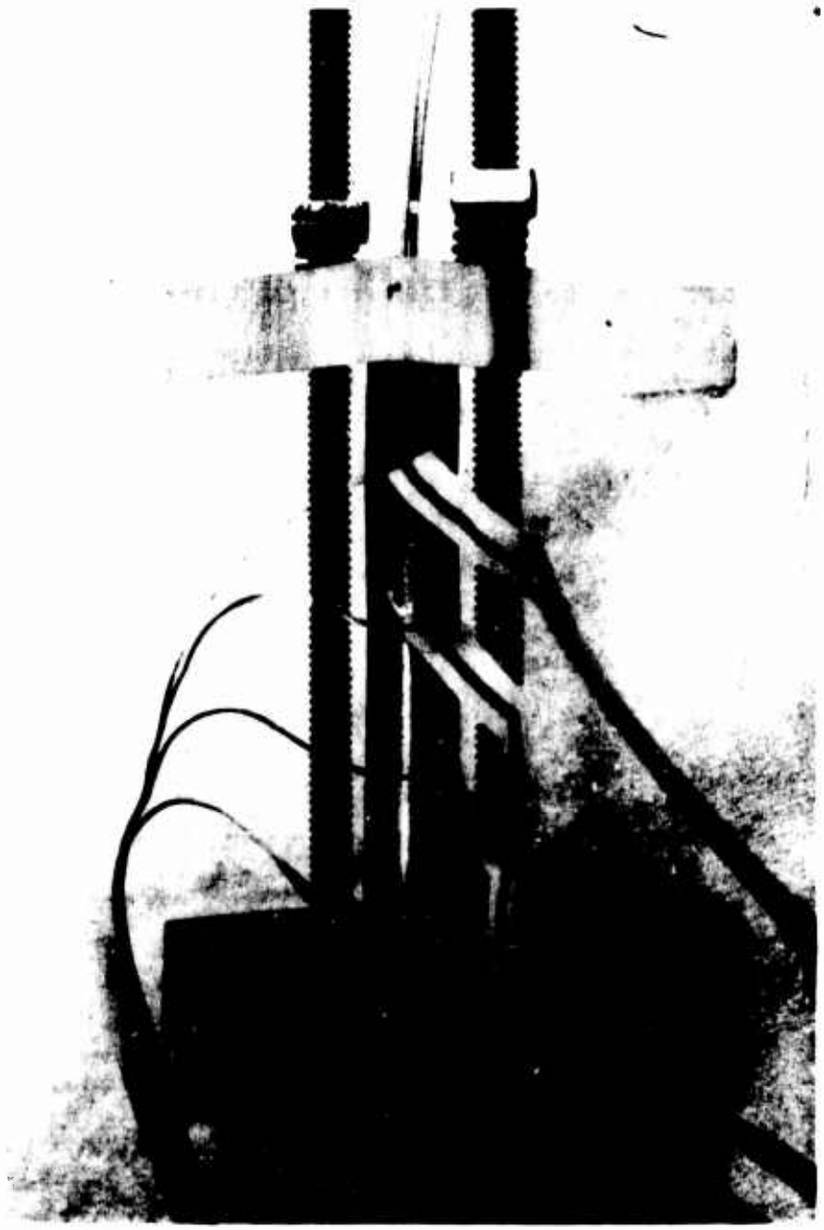
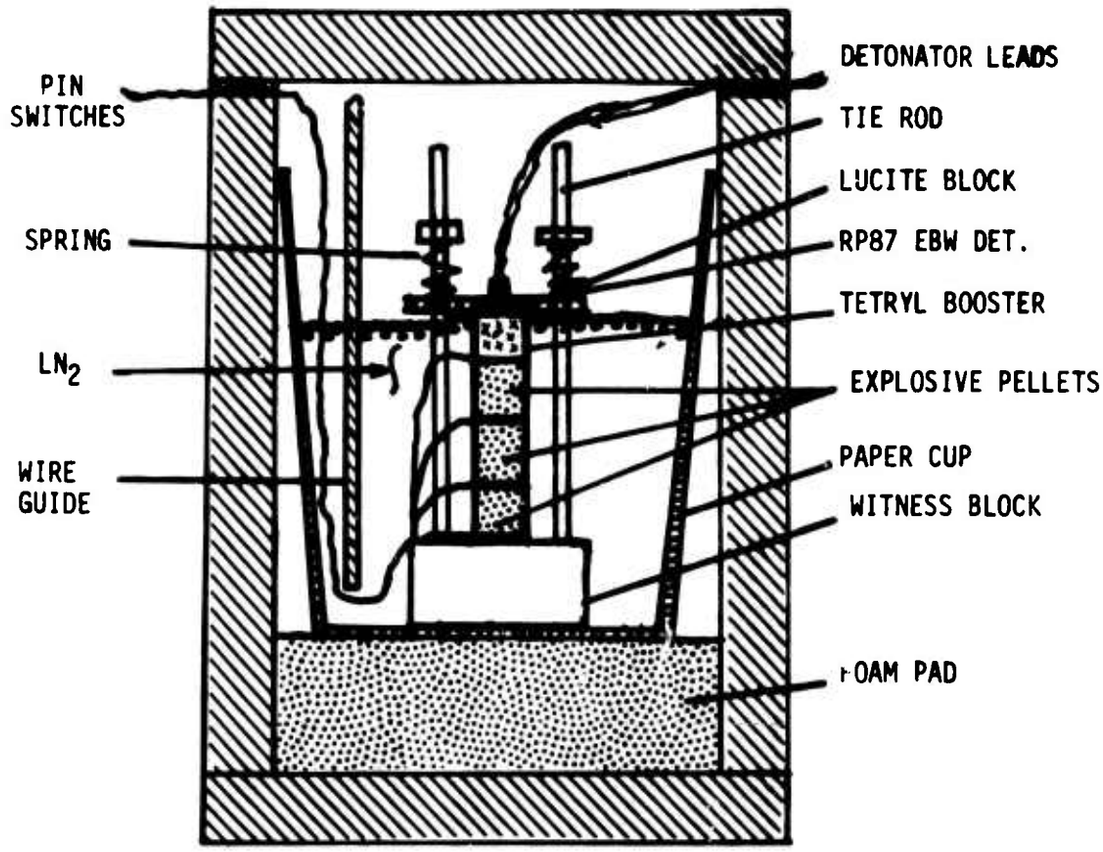


FIGURE 10. Mock-Up of Detonation Velocity Test with Unconfined TNT Pellets.



WITNESS BLOCK - 2" X 2 1/2" X 1" THICK

CHAMBER - 6 1/2" O.D., 5" I.D., 3/4" THICK END PLATES
HELD ON WITH 4 - 3/8" BOLTS

FIGURE 11. Detonation Velocity Test in LN_2 with Unconfined TNT Pellets.

irradiating explosives in a nuclear reactor (4,5,6) a triple-walled firing chamber was developed.

Normally explosive samples 3/8" diameter and 3" long were fired and a 1" OD brass sleeve located in a polyethylene foam tube which centered the explosive charge inside a 2 9/16" ID, 3 1/2" OD steel pipe. This confined the brass fragments and protects the velocity pin wires. A 1/2" thick 2" diameter aluminum disc was placed under the charge and a 1/8" thick 2" diameter aluminum disc placed on top of the explosive assembly. Each end of the 3 1/2" OD pipe was plugged with 2" of polyethylene foam. These steps were taken to protect the covers of the inner firing chamber from fragments.

The inner firing chamber was a steel pipe 6" OD, 4 9/16" ID and 7 3/4" long. The ends were closed with 1/2" thick steel plates bolted with four 3/8"-16 high strength steel cap screws 1" long. The leads from the explosive assembly were funneled through a groove on the top of the inner firing chamber pipe and the same on the outer firing chamber. The outer firing chamber consisted of a steel pipe 12 3/4" OD, 11 1/4" ID (12" schedule 80) which is 18" long. At the bottom of the outer chamber are 3" of foam upon which the inner firing chamber was supported. On top of the inner firing chamber a 2" layer of foam was

installed. Both ends of the outer firing chamber consisted of 1/2" steel thick plates bolted onto the outer pipe with six 3/8"-16 high strength steel cap screws 1" long.

A sketch of the firing chamber is shown in Figure 12 and photographs depicting different points in the testing procedure are in Figure 13.

The firing chamber was designed for a maximum of 12 grams of explosive in a brass sleeve. To verify the design of the chamber assembly a proof test of 125% of the designed amount of explosive was conducted. The grooves for the wires also served as vents for the escaping gases. Only a weak thud and a hiss of escaping gas was heard after the explosive was detonated remotely. When fired in the LN_2 environment a stronger thud and more gas was heard to escape.

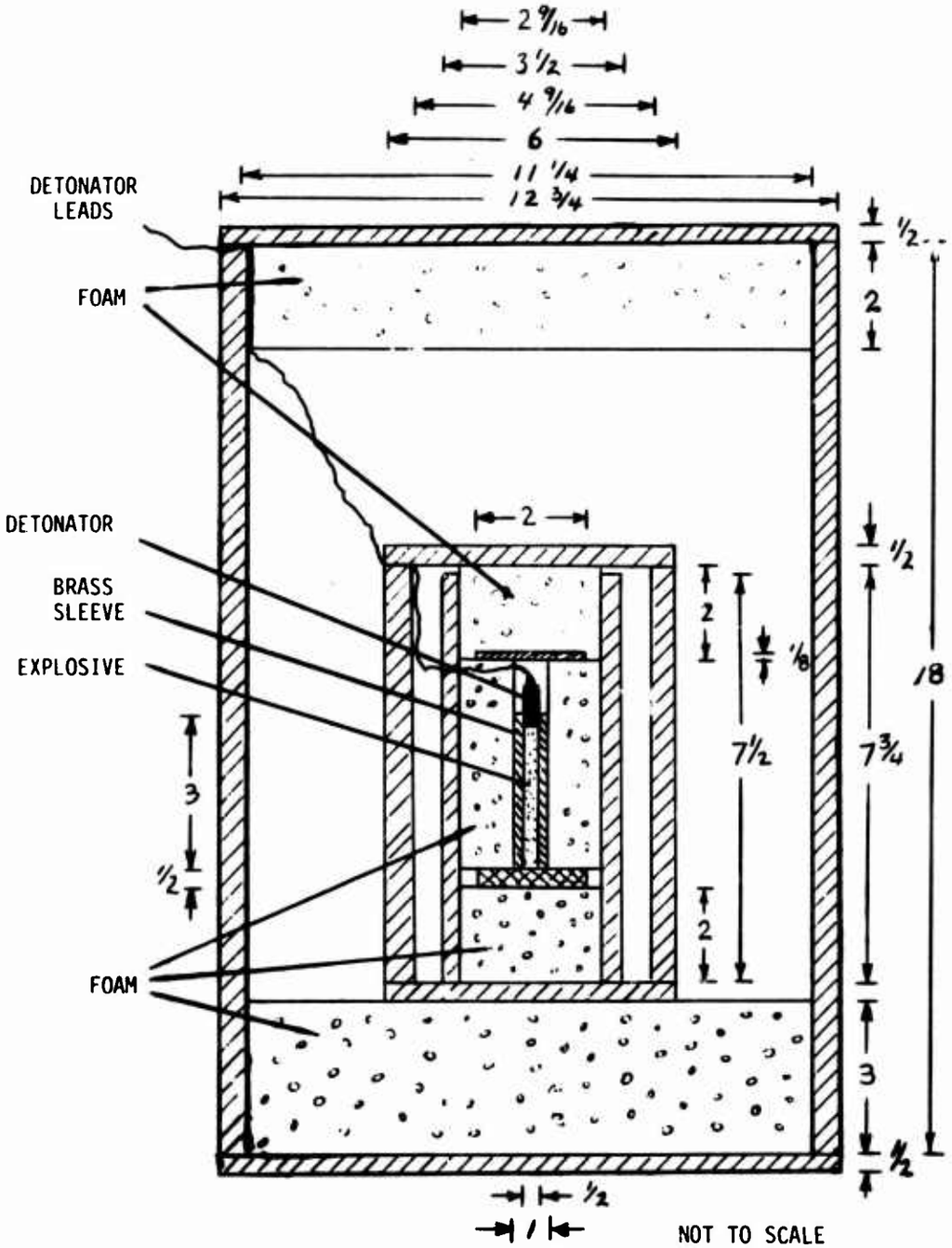


FIGURE 12. Sketch of Firing Chamber.

FIGURE 13. Three Views of Firing Chamber Set-Up.

RESULTS AND DISCUSSION

1. DTA Results

The thermograms of the DTA investigation are shown in Figures 14 through 19 and the results are listed in Table 2. Figure 14 is the regular DTA for TNT at a heating rate of 20°C/min. which was used for comparisons.

The different heating rates were used, the 20°C/min, the 5°C/min and the slower heating rate was used to try and yield some changes, however small. The TNT samples were analyzed under two different conditions. In one case the TNT was quick-cooled with LN_2. At the same time N_2 gas was flowing over the heating block. Most of the measurements were started at -100°C (173K). In the second case the TNT was brought up to the melting point and the quick-cooled in LN_2.

TNT melts over a range of several degrees with the range depending on the purity. Double melting endotherms can be evident depending on the preparation of the sample. In pure TNT several investigations (7,8) have reported polymorphic forms grown from the melt. In commercial grades of TNT the broad endotherm caused by impurities may prevent the observation of these forms. However, in both cases, when the commercial grade TNT was either directly quick-cooled in LN_2 or melted and then quick-cooled, a

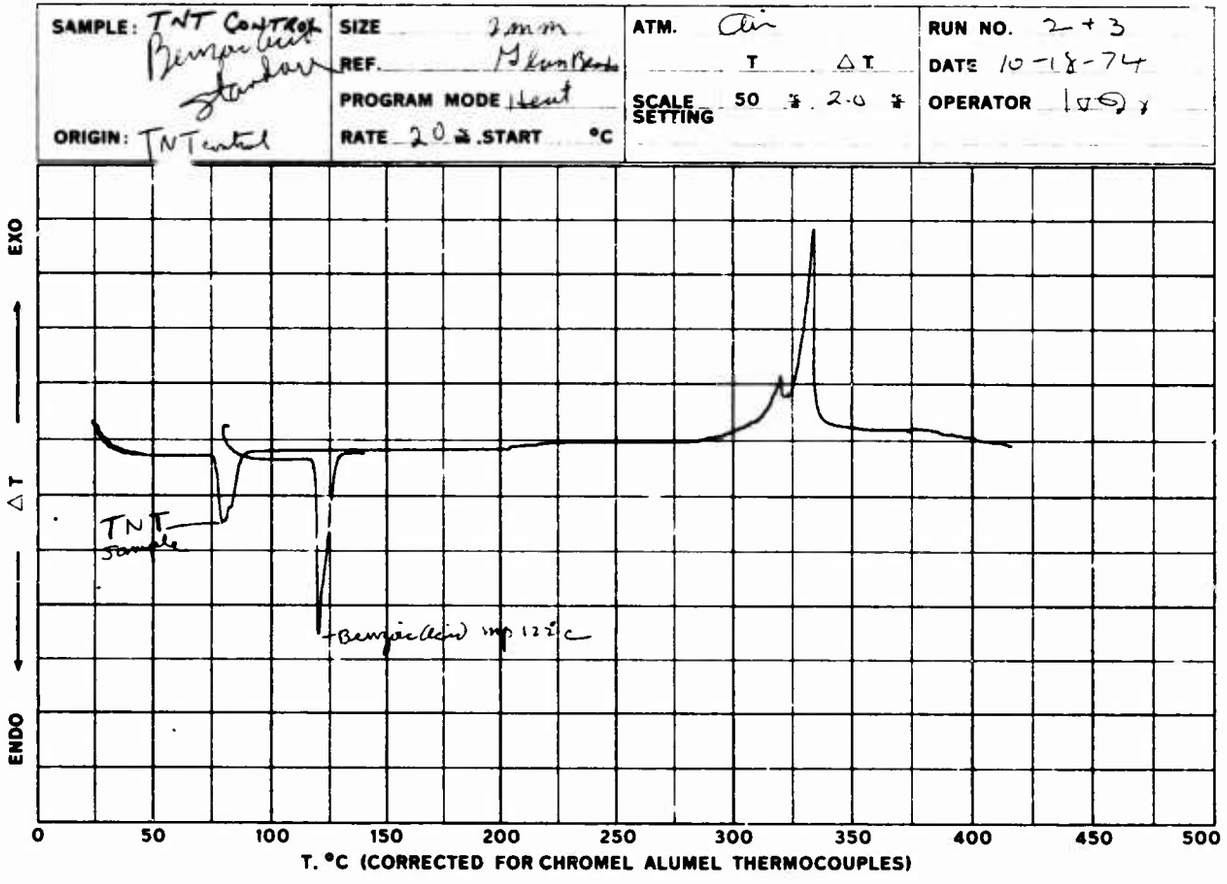


FIGURE 14. Standard DTA of TNT.

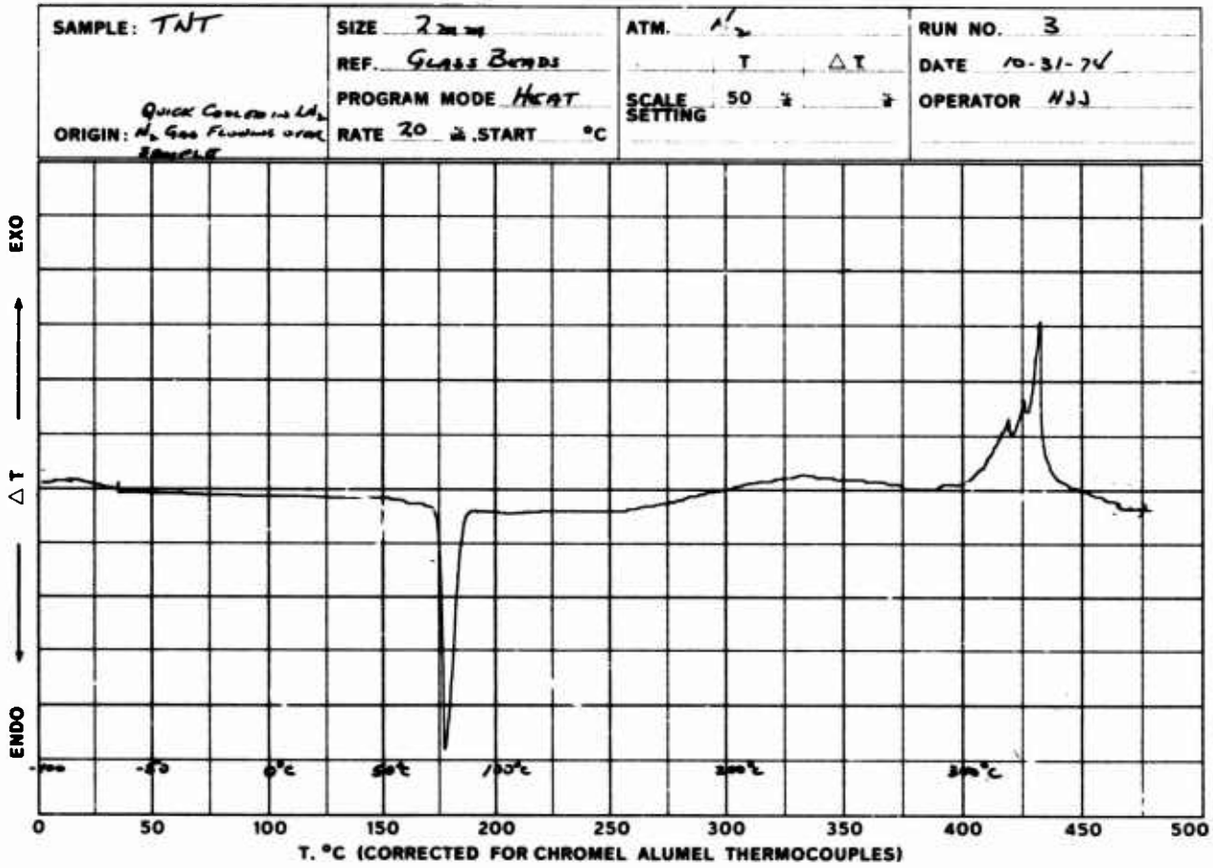


FIGURE 15. DTA of TNT Quick-Cooled in LN_2 with 20°C/min Heating Rate.

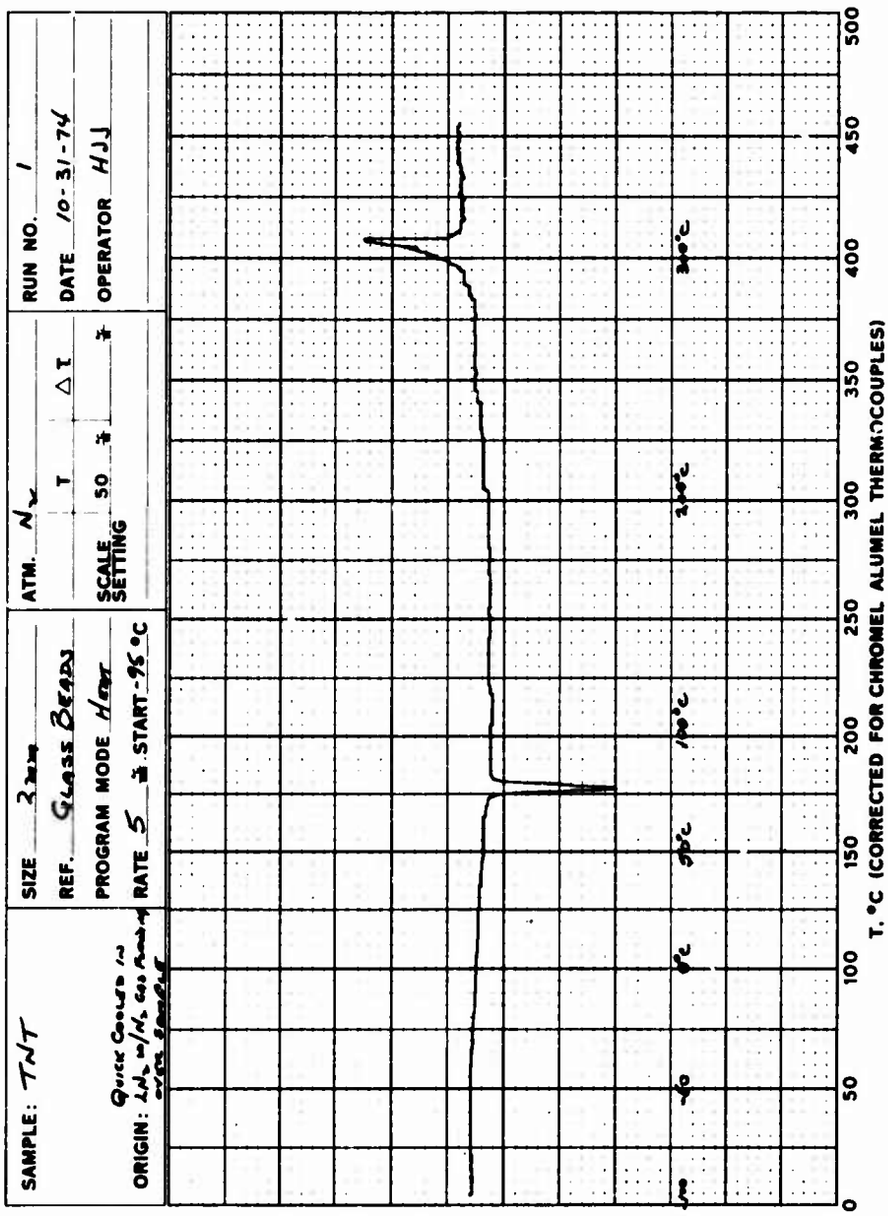


FIGURE 16. DTA of TNT Quick-Cooled in LN_2 with 5°C/min Heating Rate.

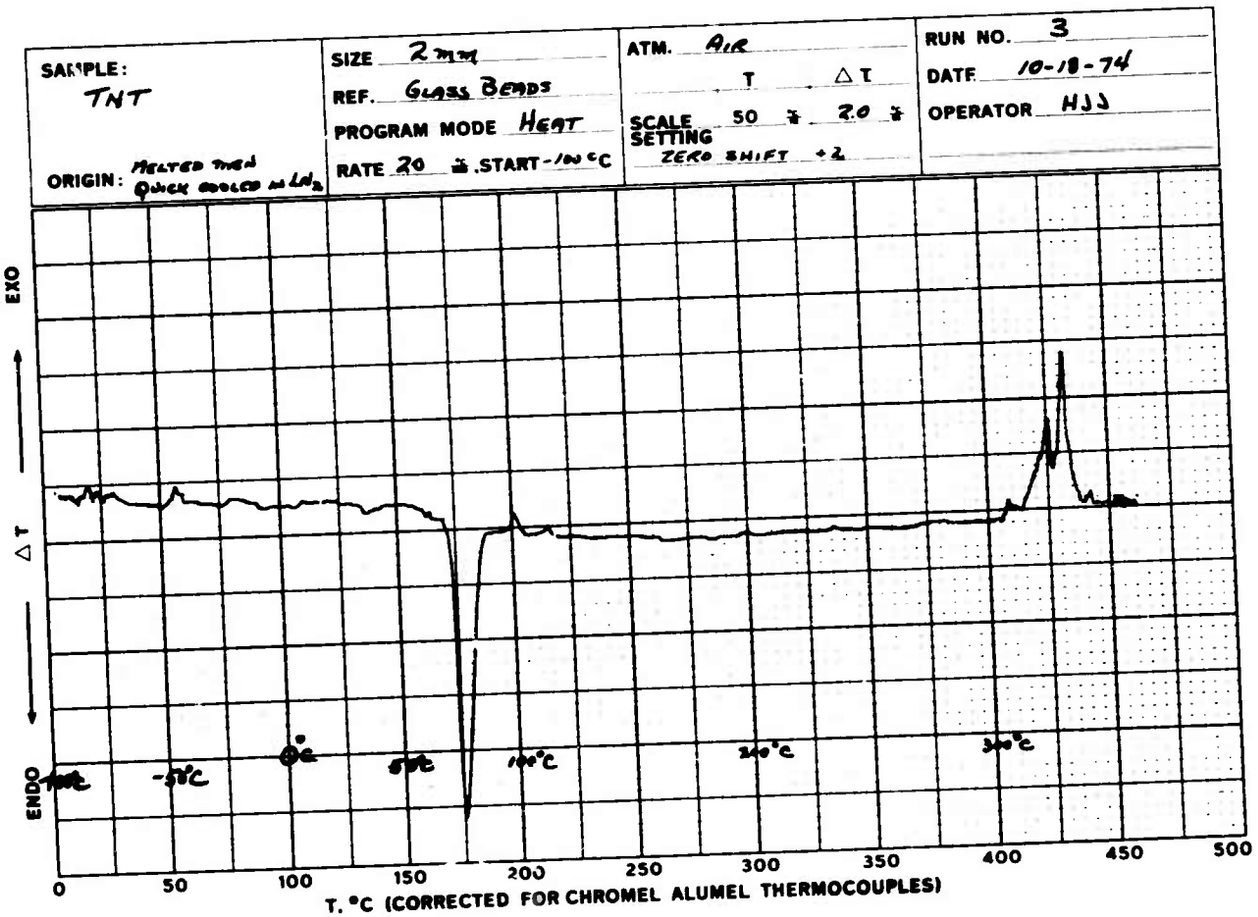


FIGURE 17. DTA of TNT-Melted then Quick-Cooled in LN_2 with 20°C/min Heating Rate.

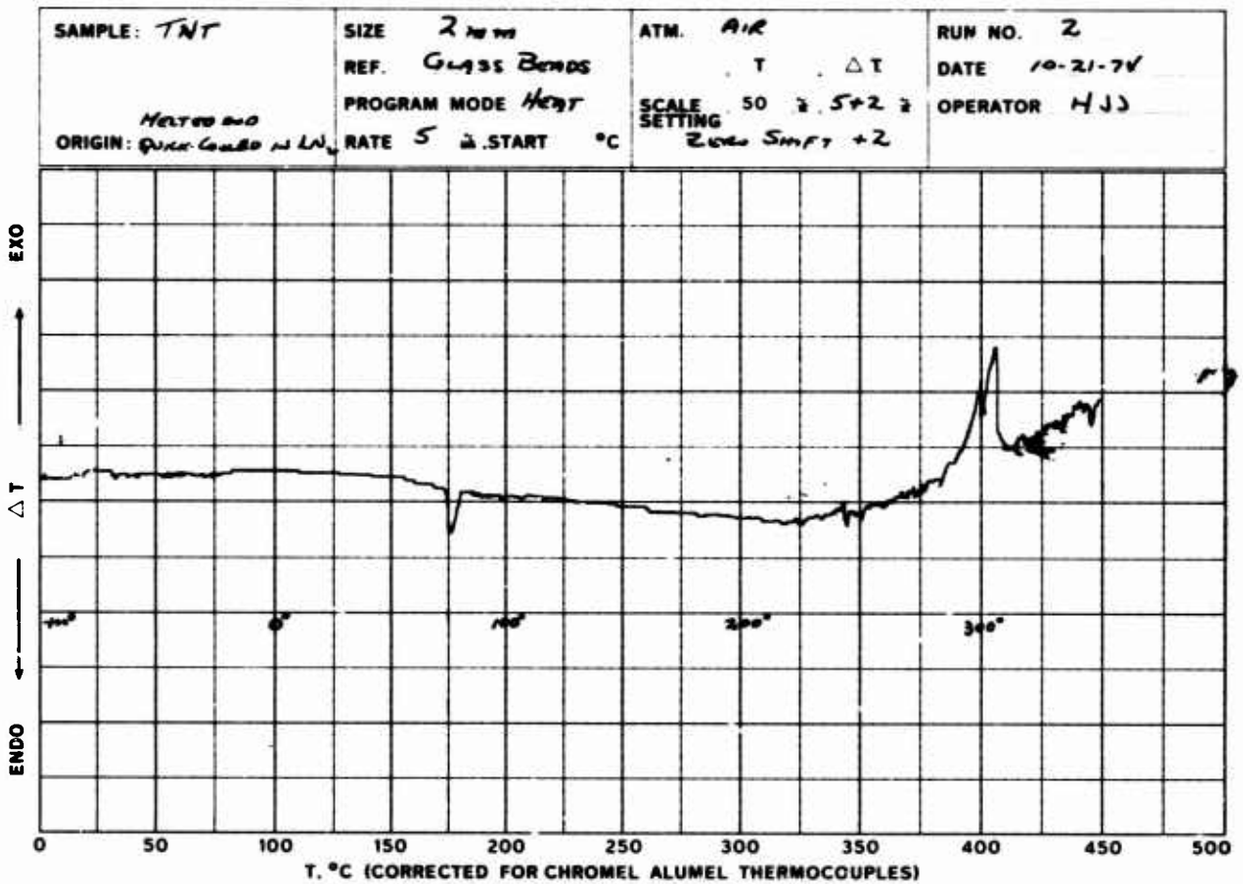


FIGURE 18. DTA of TNT-Melted and Quick-Cooled in LN_2 with 5°C/min Heating Rate.

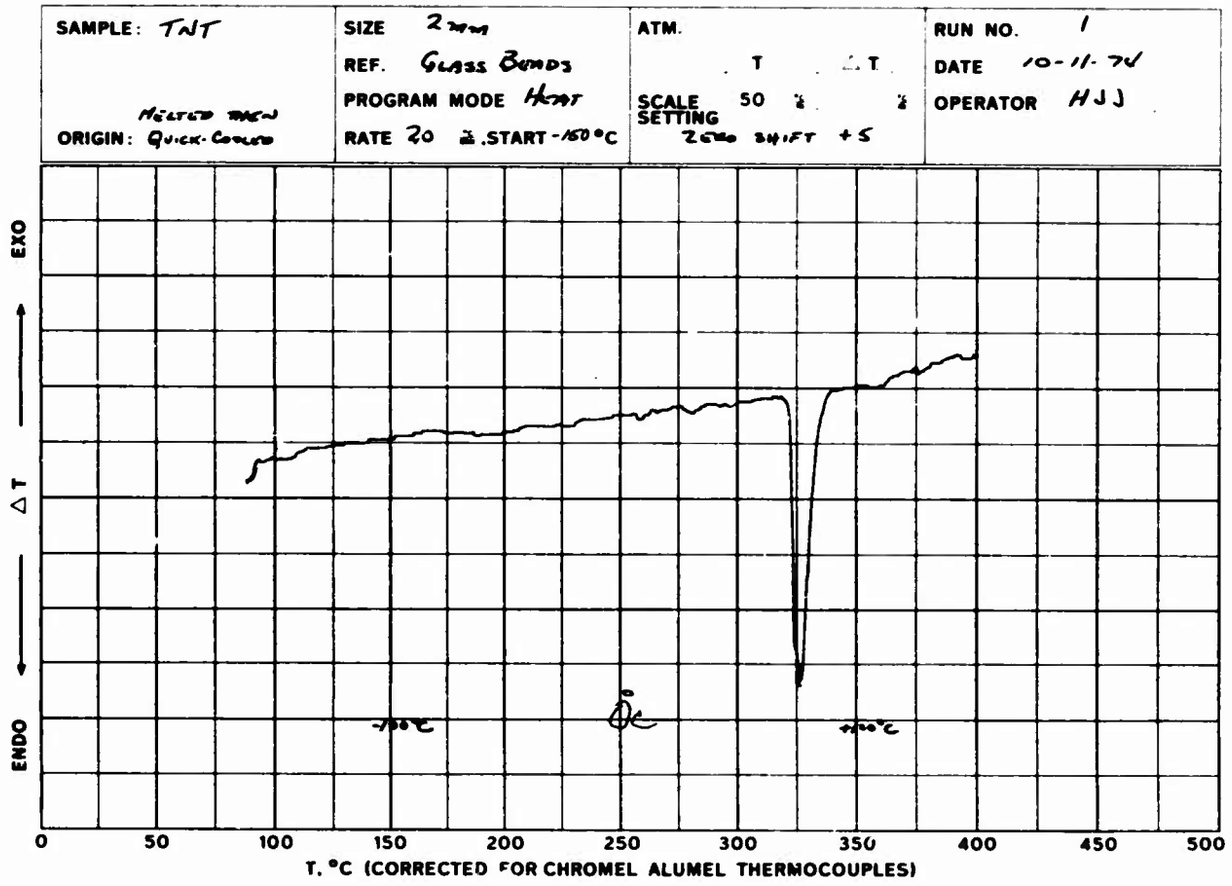


FIGURE 19. DTA of TNT-Melted and Quick-Cooled in LN_2 in -150°C to 150°C Range.

TABLE 2

DTA Result for TNT

DTA Thermogram	Heating Rate	Temperature Range	Start Endotherm	Peak Endotherm	Start Exotherm	Peak Exotherm	Notes
Fig. 14	20°C/min	+25°-+415°C	75°C	80°C	285°C	334°C	
Fig. 15	20°C/min	-100°-+375°C	68°C	77°C	∿290°C	332°C	Quick-cooled
Fig. 16	5°C/min	-100°-+350°C	65°C	77°C	∿283°C	307°C	Quick-cooled
Fig. 17	20°C/min	-100°-+350°C	65°C	76°C	∿306°C	332°C	Melted then quick-cooled
Fig. 18	5°C/min	-100°-+350°C	68°C	76°C	∿250°C	306°C	Melted then quick-cooled
Fig. 19	20°C/min	-150°-+150°C	68°C	76°C	-	-	Melted then quick-cooled

slight shift was noticed in the peak endotherm from 80°C to about 76°-77°C.

The DTA equipment was not sensitive enough to detect the second order phase reported by Heberlein (9) at 61.3°C (211.7K) for an orthorhombic crystal of α-trinitrotoluene. The DTA thermograms are relatively smooth from about -150°C (123K) up to the melting point of TNT which seem to indicate that for commercial TNT powder or flake do not show any significant changes in that temperature range. Actually the only difference in the entire DTA thermogram is the slight changes in the melting point presumably caused by the quick-cooling in LN_2

2. DSC Results

The DSC also was used to determine if any exothermic or endothermic changes occurred with TNT at low temperatures. Runs were made on TNT samples from -150°C (123K) to above melting and up to the endotherm. No changes were evident. The DSC results indicate that LN_2 did not affect the thermal stability of the TNT samples.

3. TMA Results

A brief survey of the literature revealed very little thermal expansion data on pressed and cast TNT pellets below -40°C (233K). Eubank et al (10) obtained data on

single crystals in the temperature range 20° to 70°C (293-343K) while Heberlein obtained data on single orthorhombic crystals of -TNT between -196° to 0°C (77-273K).

For pressed TNT pellets Forsyth (11) obtained a coefficient of linear expansion in the temperature range -10° to 60°C (263-333K). Also reported was the phenomenon of irreversible expansion upon heating. This was also verified in the present work in the range -100° to 78°C (173-351K).

In this program the samples were 1/4 x 1/4 inch pressed and cast TNT pellets. The TMA equipment was calibrated against an aluminum standard and the results are listed in Table 3. The equipment only permitted making measurements up from -100°C (173K).

Special emphasis was taken in the measurements in order to see if any transitions could be detected. Normally first-order transitions such as crystal-crystal and melting show a characteristic behavior in TMA. However the second-order phase chance at 211.7K as reported by Heberlein (9) for an orthorhombic α-TNT crystal could not be detected in either type of pellet which was not surprising.

TABLE 3

Coefficient of Linear Expansion of TNT
Cast and Pressed - by TMA

Program Rate - 10°C/min
Gas Flow - 60cc/min
Starting Environment - LN_2

Sample	Temperature Range		10^6 in/in/°C*
TNT-Cast #1	-100° to 78°C		57.50
TNT-Cast #2	-100° to 73°C		57.10
TNT-Cast #3	-100° to 78°C		51.82
TNT-Cast #4	-100° to 78°C		66.30
TNT-Cast #5	-100° to 78°C		64.09
		Ave.	59.36
TNT-Pressed #1	-100° to 68°C		64.74
TNT-Pressed #2	-100° to 78°C		67.10
TNT-Pressed #3	-100° to 78°C		67.93
TNT-Pressed #4	-100° to 78°C		65.72
TNT-Pressed #5	-100° to 78°C		67.24
TNT-Pressed #6	-100° to 68°C		63.82
		Ave.	66.09

*Calibrated to an aluminum standard.

The results in Table 3 indicate that the average thermal expansion in the pressed pellets is slightly higher than in the cast pellets but that the range in the cast pellets was larger.

These readings were obtained only in the expansion mode or from -100° to 78°C. An effort was made to measure again the thermal expansion with the same pellets but in every case the cast and pressed pellets were larger and the expansion was not as great as the original measurement. The temperature cycling probably caused a permanent expansion which was brought about by recrystallization (11).

The results listed in Table 3 are in agreement with the results published by others (9, 10, 11, 10). These results were used in the calculations for determining the densities of the samples fired for the detonation velocities of TNT in liquid nitrogen.

4. Impact Test Results

In this program an effort was made to obtain a complete impact sensitivity curve. Since a restriction of 36" limited the height of the drop weight, the weight was increased up from 2 kg to 3, 5, 6 and finally 8 kg.

The increase in the mass and size of the drop weight caused the maximum drop height to be lowered from

33 inches (84.82 cm) for 2 kg, to 25 inches (63.50 cm) for 5 kg, to 24 inches (60.96 cm) for 6 kg, and to 16 inches (40.64 cm) for 8 kg.

In order to maintain the same conditions and to minimize the variables the ambient control tests were performed in the dry state using the modified fixture and then tested in the LN_2 environment. A "fire" or "go" is defined as any audible or visible evidence of decomposition.

The control values obtained with the cryogenic fixture in the dry state were compared to those obtained with the standard Picatinny method. In all cases the values were higher with the cryogenic fixtures than those with the standard. This is attributed to the modified fixture not being imbedded in the concrete base as with the standard Picatinny impact tester. However, since trends are to be determined, the relative values using the same parameters would indicate any changes with the addition of LN_2.

The results for each of the drop weights are listed in Table 4. At each of the heights 20 samples were tested. In order to utilize the data for all the drop weights the term "work = weight x height" was used in plotting against the percentage of "fires". In this way plots can be made by one or all the drop weights.

TABLE 4

The Effect of Liquid Nitrogen on the Impact Testing of TNT

Drop Weight (kg)	Height of Fall (in)	(cm)	Work (kg-cm)	% Fire Dry	% Fire in LN_2
2	24	60.96	121.9	0	0
2	28	71.12	142.1	0	0
2	30	76.20	152.4	5	5
2	32	81.28	162.5	5	0
3	24	60.96	182.9	5	5
3	33	84.82	254.5	25	5
5	11	27.94	139.7	0	0
5	12	30.48	152.4	0	0
5	13	33.02	165.1	5	0
5	14	35.56	177.8	15	5
5	15	38.10	190.5	25	0
5	16	40.64	203.2	15	5
5	17	43.18	215.9	25	0
5	18	45.72	228.6	15	5
5	19	48.26	241.3	25	10
5	20	50.80	254.0	20	25
5	21	53.34	266.7	30	30
5	22	55.88	279.4	50	40
5	23	58.42	292.1	45	25
	24	60.96	305.7	45	25
5	25	63.50	317.5	50	45
6	10	25.40	152.4	0	0
6	12	30.48	182.9	10	5
6	14	35.56	213.4	15	15
6	16	40.64	243.8	10	15
6	18	45.72	284.3	50	30
6	20	50.80	304.8	40	30
6	22	55.88	335.3	50	40
6	24	60.96	365.8	75	55
8	8	20.32	162.6	0	0
8	10	25.40	193.2	5	0
8	12	30.48	243.8	20	20
8	14	35.56	294.5	45	30
8	16	40.64	325.1	40	30

NOTE: Each height represents 20 samples tested.

The data were subjected to statistical analysis by the method proposed by Kemmey (13). That method combines the χ^2 (chi-square) or goodness-of-fit test with the Karber test to provide a means to rate the materials in terms of relative sensitivity. In the χ^2 test the 95% level of confidence was chosen, and the degrees of freedom correspond to the number of drop heights, or in this case, work values (kg-cm).

Comparisons between samples could not have been obtained if the measurements had not been made at the same drop heights or work values. Even under these circumstances the available computer program will permit the Karber statistical analysis to be performed and give a graphical plot of the percent fire versus the work value or drop height.

A complete firing curve could not be obtained due to the design of the impact tester. In the cases where the χ^2 test could not be applied differences in sample behavior due to the LN_2 were shown graphically in the computer printouts.

The analysis first was done relative to the data for each of the drop weights and then all the data. Due to the minimum amount of data the 2 and 3 kg data were not analyzed. For the 8 kg data the computer code indicated that the distribution was such that an adequate test could not be made.

Analyses were obtained for the 5 kg, 6 kg and the combined data. The 5 kg data analysis indicated that the samples were different with a certainty of 95%. The 6 kg data could not be certain with a 95% confidence level although the graph showed a definite difference. However the combined data showed that the samples were different with a 95% confidence level.

The reason that the 6 kg data was not in agreement with the other data was due to the incomplete distribution with the preponderance of data below the 50% point. Another reason was that the Karber test could not be applied to obtain the mean critical work value (or drop height) although graphically it could be extrapolated.

Estimates of the mean critical work value at the 50% fire point were obtained from the graphs. With the 5 kg data the control value (22°C and dry conditions) was 315 kg-cm and in LN_2 330 kg cm; for 6 kg cm; for all the data combined the control was 300 kg cm and in LN_2 343 kg cm.

A review of the data indicates that TNT at LN_2 temperature is less sensitive to impact at drop weights up to 8 kg. However, the trend indicates that if enough work is applied (i.e. 20 kg at 50 cm) the application of LN_2 will not effect the initiation due to impact.

5. Detonation Velocity and Plate Dent Results

The detonation velocity and plate dent tests were conducted on pressed and cast, confined and unconfined TNT pellets under ambient and LN_2 conditions. The results are listed in Table 5.

Due to the effects of LN_2 to detonators as reported previously (3) and by Trott (15) extra care was taken to prevent the submersion of the initiating detonator in the LN_2 during the tests. In the previous work (3) it was shown that unless maximum current is used failures can be expected when testing detonators submerged in LN_2.

The effect of LN_2 on the density of the TNT pellets was corrected by using the data generated with the TMA.

Unconfined TNT pellets shrink at liquid nitrogen temperature (-195.8°C). The distance between foil switches was therefore reduced as follows:

$$L_{LN_2} = L_{20°C} \left(1 - \frac{\Delta L}{L \Delta T} \Delta T\right)$$

Where $\Delta T = 215.8°C$

$\frac{\Delta L}{L \Delta T} = 66.08 \times 10^{-6}$ in/in/°C for pressed TNT

and $\frac{\Delta}{L \Delta T} = 59.26 \times 10^{-6}$ in/in/°C for cast TNT.

TABLE 5

Effect of Liquid Nitrogen on Detonation Velocity of TNT

Dia.	No. and Type	Confinement	Environment	Ambient	LN_2	Det Vel mm/μsec	Plate Dent
1/4"	Pressed #1	Al(.028" wall)	Ambient	1.60	-	6.957(1)	
	#2					6.917(2)	
	#3					6.821	
	#4					6.751	
	#5					6.956	
	#6					6.984(4)	
	#7					6.512	
	#8					7.025	
	#9					6.984	
					AVE	6.857	
1/4"	Pressed #11	Al(.028" wall)	LN_2	1.60	1.64	6.757	Shattered
	#12					6.644	"
	#13					6.737	"
	#14					7.063	"
					AVE of 4	6.800	
				(w/o #14)	" 3	6.710	
1/4"	Cast	Al(.028" wall)	Ambient	1.60	-	Fired low order (6 tests)	
1/4"	Cast	Al(.028" wall)	LN_2	1.60	1.64	Fired low order (3 tests)	

TABLE 5 (contd)

Dia.	No. and Type	Confinement	Environment	Ambient	LN_2	Det Vel mm/μsec	Plate Dent
3/8"	Pressed #1	Brass(.312wall)	Ambient	1.59	-	6.904 (10)	-
	#2			1.59	-	6.945 (6)	.070
	#8			1.58	-	6.908 (5)	.067
					AVE	6.919	
3/8"	Pressed #43	Brass(.312"wall)	LN_2	1.58	1.62	6.605 (4)	.065
	#44					6.735 (4)	.077
	#45					6.617 (3)	.070
	#46					6.618 (5)	.060
					AVE	6.644	AVE.068
1/2"	Pressed #11	None	Ambient	1.58	-	6.772 (3)	.072
	#12					6.553 (3)	.068
	#13					6.789 (1)	.069
					AVE	6.705	AVE.070
1/2"	Pressed #14	None	LN_2	1.58	1.64	6.959 (1)	.055
	#1					6.837 (3)	.053
	#6					6.658 (3)	.036
	#2					6.888 (2)	.035
	#3					6.764 (2)	.038
	#4					no record	.038
	#5					no record	.037
					AVE	6.821	AVE.042
1/2"	Cast	None	Ambient	1.58	- DID NOT FULLY DETONATE		
1/2"	Cast	None	LN_2	1.58	1.64 DID NOT FULLY DETONATE		

NOTE: (number) = number of points averaged.

Confined charges had the timing pins imbedded in the confining tube, and so their spacing would depend on the contraction of the tube. For aluminum, $\frac{\Delta L}{L\Delta T} = 12 \times 10^{-6}$ in/in/°F, and for brass, 17.5×10^{-6} in/in/°F.

In addition to the effects on switch spacings the density of the TNT increased as follows:

$$P_{LN_2} = \frac{P_{20}}{(1-\frac{\Delta L}{L\Delta T}\ \Delta T)^3}$$

Therefore a TNT pellet having a density of 1.58 gm/cc at 20°C would have a density of 1.64 gm/cc at LN_2 temperature. The corrections are noted in the table.

The results on the lightly confined 1/4" pressed TNT pellets as shown in Table 5 show a detonation velocity value of 6.857 mm/μsec under ambient conditions and 6.800 m/μsec in LN_2. The ambient values are consistent with those reported by Urizar et al (16) but the slight decrease in the detonation velocity in LN_2 was surprising. The plate dent values were not obtained with this test since all of the dent plates immersed in LN_2 shattered when the shock impinged on the plate.

The lightly confined 1/4" cast TNT pellets did not produce any results that are reportable. In several cases the reaction did not propagate more than one-half the length of the TNT pellets either in air or LN_2. Also

several tests with the pressed pellets showed the same results indicating that the 1/4" lightly confined test may be adequate for the pressed but below the failure diameter of the cast TNT pellets.

The heavily confined 3/8" pressed TNT in the brass cylinder with a .312" wall produced consistent results. However, a small change had to be made due to the experimental set-up.

Since the sensors are at the edge of the charge, and the initiation point is in the center, as correction due to the pin spacings is required. The corrections in the following table are needed.

3/8" Diameter Detcnation Velocity Corrections

Pin Nos.	Correction Factor	Pin Nos.	Correction Factors
0-1	0.9768	0-6	0.9916
1-2	0.9888	1-6	0.9949
2-3	0.9933	2-6	0.9960
3-4	0.9957	3-6	0.9969
5-6	0.9979	4-6	0.9973

These corrections were obtained from the relationships:

$$D_{true} = \frac{d_j - d_i}{h_j - h_i} D_{observe}$$

Where: $d = (.1875)^2 + h^2$

d = true distance

h = distance along side of tube

and subscripts refer to successive points.

The corrected results are shown in Table 5. In this test perhaps due to the paucity of data the detonation velocity was reduced from an average of 6.919 mm/μsec in air to 6.44 in LN_2. This trend was evidenced also with the 1/4" lightly confined pressed pellets.

The plate dent results seem to indicate the same trend. However the 1 1/2" diameter x 3/4" thick plates cracked when tested in LN_2 as shown in Figure 20. One plate split in half (Figure 21). The stresses can be seen in the cross-section of the plate which were caused by the compressive shock in LN_2.

The 1/2" unconfined pressed TNT pellets in ambient and LN_2 produced detonation velocities which were opposite to the trend obtained with the lightly and heavily confined TNT pellets. In air the average velocity was 6.705 mm/μsec while in LN_2 a slight increase was noted at 6.821 mm sec. Here more data may be required to see if there is a real difference.

The plate dent results did show a definite decrease due to the LN_2. This can be seen in Figure 22.

The tests on 1/2" unconfined cast TNT pellets proved disappointing. Full detonation was never achieved in either air or in LN_2. The results can be seen in Figure 23 for several of the tests. The critical diameter of cast TNT,

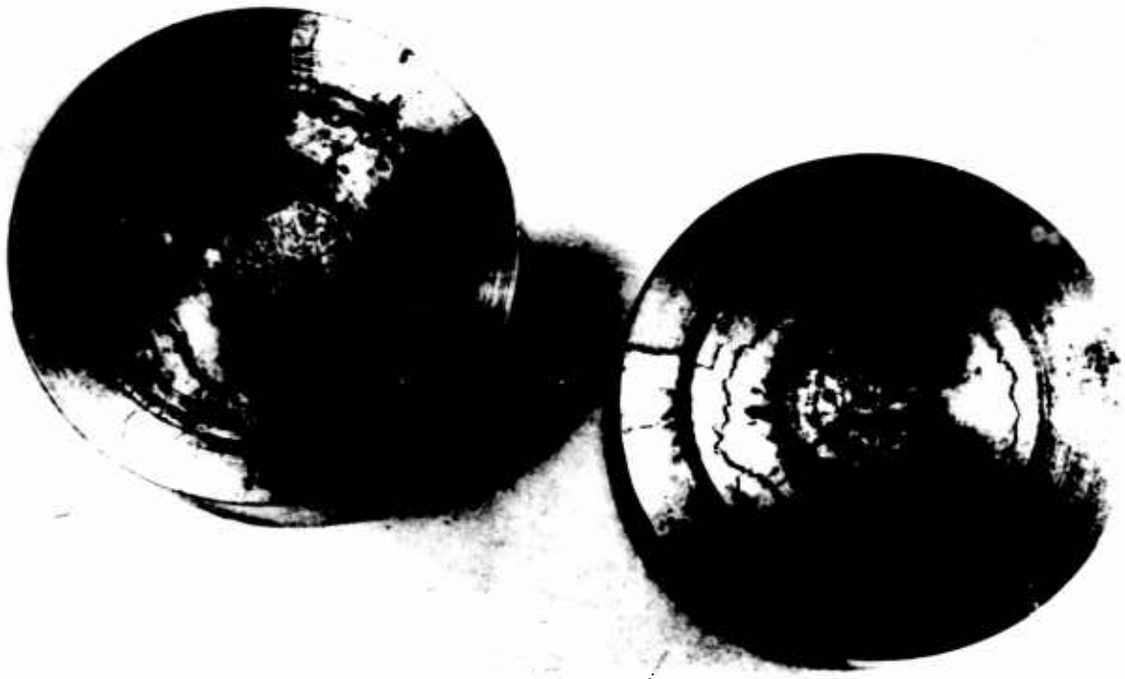
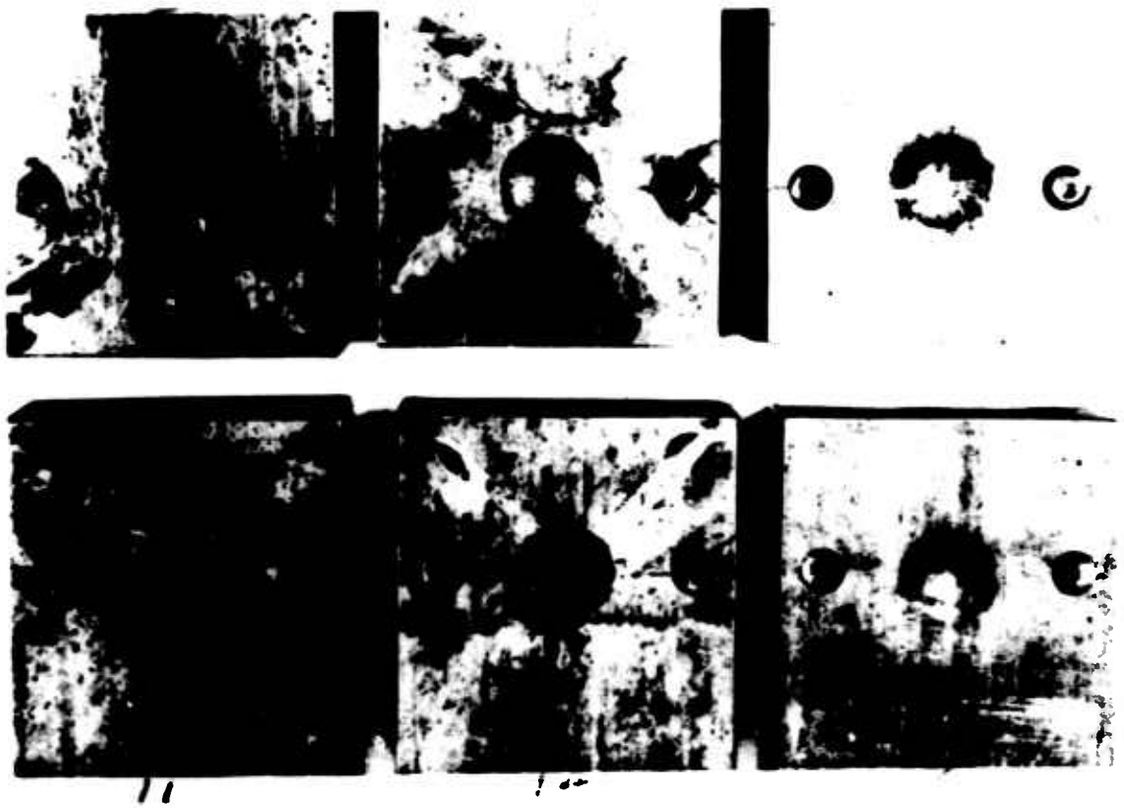


FIGURE 20. Plate Dent Results on 3/8" Heavily Confined Pressed TNT Pellets in LN_2.

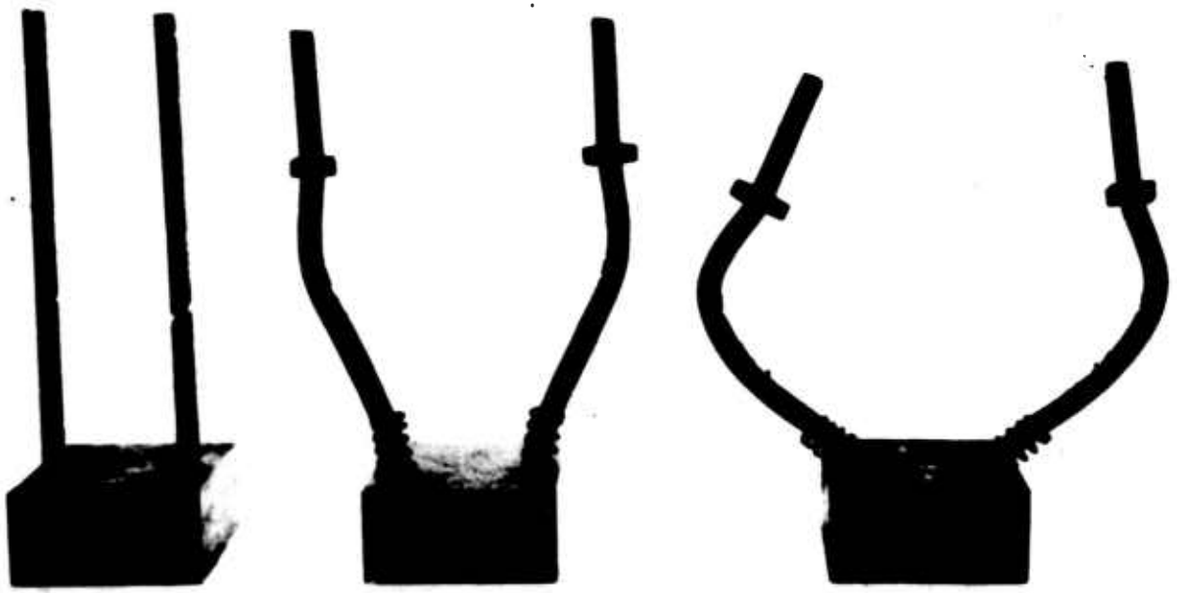
FIGURE 21. Plate Dent Shattering of 3/8" Heavily Confined Pressed TNT Pellets in LN_2.



(a) (b) (c)

FIGURE 22. Plate Dent Results on 1/2" Unconfined Pressed TNT Pellets

(a) in air - 72, 68 mil dents
(b) in LN_2 - 52, 54 mil dents
(c) in LN_2 - 37, 38 mil dents



(a) (b) (c)

FIGURE 23. Results of 1/2" Unconfined Cast TNT Pellets

(a) No detonation in LN_2.
(b) 2/3 detonation in LN_2.
(c) 5/6 detonation in LN_2.

confined or unconfined, is not the same as pressed TNT. Both are a function of temperature but the cast TNT critical diameter seems to have been affected more by the LN_2 environment. Although a value of 31 mm has been reported (17) for cast unconfined TNT, the effect of temperature and density were not clear. Also information seems to be lacking for the critical diameter for confined cast TNT. These factors prompted the tests as conducted.

SUMMARY AND RECOMMENDATIONS

In summarizing the data obtained in this program the following statements can be made:

1. In the temperature range of -150°C to 80°C the DTA, DSC and TMA methods did not reveal any significant or new changes (i.e. phase transitions, crystal-crystal, etc.) in military-grade TNT.
2. In the temperature range -100°C to 78°C the average coefficient of linear expansion for cast TNT pellets by the TMA method is 59.36×10^{-6} in/in/°C. For pressed TNT pellets the average coefficient of linear expansion is 66.09×10^{-6} in/in/°C. For cast and pressed TNT pellets the dimensions and densities were the same.
3. In the impact sensitivity test with the modified fixture the results indicate that with drop weights up to

8 kg TNT is less sensitive in LN_2 with a 95% confidence level. (Above that the trend indicates that if enough work or energy is put into the impact the LN_2 environment will not have any effect.)

4. In the miniature detonation velocity tests developed only the pressed TNT pellets gave acceptable results:

a. The 1/4" lightly confined pressed TNT pellets displayed a slightly lower detonation velocity when tested in LN_2 (6.857 to 6.800 mm/μsec).

b. The 3/8" highly confined pressed TNT pellets produced a much lower detonation rate when tested in LN_2 (6.919 to 6.644 mm/μsec).

c. However the 1/2" unconfined pressed TNT pellets reversed the trend obtained with the confined samples. When tested in LN_2 a higher detonation velocity was obtained (6.705 to 6.821 mm/μsec). A comparison of the detonation rates of the confined vs unconfined pressed pellets reveals that when tested under ambient conditions the detonation velocity of the unconfined pellets was lower than that for the confined pellets.

d. On an overall basis the averages of all the detonation velocities with confined and unconfined pellets reveal that the change when tested in LN_2 was less than .100 mm/μsec. (However more data is needed to substantiate this statistically.)

5. The plate dent results, although meager, indicate that with confined pressed TNT pellets similar results are obtained in LN_2 and under ambient conditions. However, a definite decrease is evident in the depth of the dent when the plate dent tests are conducted with unconfined pressed TNT pellets in LN_2.

6. No acceptable results were obtained in the detonation velocity tests with confined or unconfined cast TNT pellets. This obviously eliminated any plate dent results.

The inability to obtain detonation velocity data with cast TNT pellets by any of the methods developed indicates that larger diameter samples should be investigated. The effect of temperature on the critical diameter on confined and unconfined cast TNT pellets needs further study.

The performance of TNT under LN_2 conditions should require inputs from card gap tests, other shock initiation tests, brisance, and fragmentation velocity measurements. The plate dent test results indicate that shock effects in solids under LN_2 conditions need further study.

The minimum initiating charge of various initiating materials required to produce high order detonations in TNT while in LN_2 should be determined. This also includes studying the means of initiation (EBW, hot wire, stab, percussion, etc.) under ambient and LN_2 conditions.

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